

FORM-PTO-1390  
(Rev. 12-29-99)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

**TRANSMITTAL LETTER TO THE UNITED STATES  
DESIGNATED/ELECTED OFFICE (DO/EO/US)  
CONCERNING A FILING UNDER 35 U.S.C. 371**

003300-817

U.S. APPLICATION NO. (if known, see 37 C.F.R. 1.5)

09/913938

INTERNATIONAL APPLICATION NO.  
PCT/SE00/00066

INTERNATIONAL FILING DATE  
14 January 2000

PRIORITY DATE CLAIMED  
2 March 1999

TITLE OF INVENTION  
COPPER RECOVERY PROCESS

APPLICANT(S) FOR DO/EO/US  
ROBERT PACHOLIK and GUNNAR LIDMER

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and the PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
  - a. ☒ is transmitted herewith (required only if not transmitted by the International Bureau).
  - b. ☒ has been transmitted by the International Bureau.
  - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US)
6. ☐ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
  - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
  - b. ☐ have been transmitted by the International Bureau.
  - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
  - d. ☒ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). (Signed Declaration will follow)
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern other document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A **FIRST** preliminary amendment.
 ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☒ Other items or information: Transmittal of New FIG. 1. A certified copy of Swedish Application No. 9900748-6, filed 2 March 1999, was submitted during the international phase of the prosecution. Thus, the claim for priority has been perfected.

U.S. APPLICATION NO. (if known, see 37 CFR 1.507) <b>09/913938</b>		INTERNATIONAL APPLICATION NO. PCT/SE00/00066		ATTORNEY'S DOCKET NUMBER 003300-817	
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17. <input checked="" type="checkbox"/> The following fees are submitted:				CALCULATIONS		PTO USE ONLY	
<b>Basic National Fee (37 CFR 1.492(a)(1)-(5)):</b>  Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO ..... \$1,000.00 (960)  International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO ..... \$860.00 (970)  International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO ..... \$710.00 (958)  International preliminary examination fee paid to USPTO (37 CFR 1.482) but all claims did not satisfy provisions of PCT Article 33(1)-(4) ..... \$690.00 (956)  International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(1)-(4) ..... \$100.00 (962)							
<b>ENTER APPROPRIATE BASIC FEE AMOUNT =</b>							
Surcharge of \$130.00 (154) for furnishing the oath or declaration later than months from the earliest claimed priority date (37 CFR 1.492(e)). 20 <input type="checkbox"/> 30 <input type="checkbox"/>				\$ 1,000.00			
				\$ -			
Claims	Number Filed	Number Extra	Rate				
Total Claims	43 -20 =	23	X\$18.00 (966)	\$ 414.00			
Independent Claims	1 -3 =	0	X\$80.00 (964)	\$ -			
Multiple dependent claim(s) (if applicable)				+ \$270.00 (968)	\$ -		
<b>TOTAL OF ABOVE CALCULATIONS =</b>				\$ 1,414.00			
Reduction for 1/2 for filing by small entity, if applicable (see below).				\$ -			
<b>SUBTOTAL =</b>				\$ 1,414.00			
Processing fee of \$130.00 (156) for furnishing the English translation later than months from the earliest claimed priority date (37 CFR 1.492(f)). 20 <input type="checkbox"/> 30 <input type="checkbox"/>				\$ -			
				+			
<b>TOTAL NATIONAL FEE =</b>				\$ 1,414.00			
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 (581) per property +				\$ -			
<b>TOTAL FEES ENCLOSED =</b>				\$ 1,414.00			
				Amount to be: refunded		\$	
				charged		\$	

a. ☐ Small entity status is hereby claimed.

b. ☒ A check in the amount of \$ 1,414.00 to cover the above fees is enclosed.

c. ☐ Please charge my Deposit Account No. 02-4800 in the amount of \$ \_\_\_\_\_ to cover the above fees. A duplicate copy of this sheet is enclosed.

d. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 02-4800. A duplicate copy of this sheet is enclosed.

**NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.**

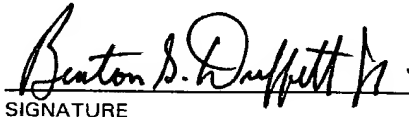
  

SEND ALL CORRESPONDENCE TO:

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Filed: August 20, 2001

  
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 Benton S. Duffett, Jr.  
 NAME  
  
22,030  
 REGISTRATION NUMBER

00015938 1100201

09/913938

JG08 Rec'd PCT/PTU 20 AUG 2007

Patent

Attorney's Docket No. 003300-817

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re Patent Application of	)	
	)	<b>BOX PCT</b>
ROBERT PACHOLIK et al.	)	<b>Attn: DO/EO/US</b>
	)	
Application No.: (unassigned)	)	Group Art Unit: (unassigned)
	)	Examiner: (unassigned)
Filed: August 20, 2001	)	
	)	
For: COPPER RECOVERY PROCESS	)	

**PRELIMINARY AMENDMENT**

Assistant Commissioner for Patents  
Washington, D.C. 20231

Sir:

This is a national phase filing of International Application No. PCT/SE00/00066,  
filed January 14, 2000.

Please amend this Application as indicated.

**IN THE ABSTRACT:**

Please add the Abstract of the Disclosure that is provided on a separate sheet.

**IN THE CLAIMS:**

Kindly replace Claims 1, 5 to 21 and 23 to 25 as follows:

1. (Amended) A process for recovering copper from an alkaline etch bath from  
an etching process in which printed boards electroplated with copper are etched with the  
alkaline etch bath and then rinsed with water, copper being removed from the alkaline etch

bath by extraction with an organic solution containing a reagent, which forms with copper a complex compound, which is extracted by the organic solution, the alkaline etch bath being recirculated to renewed etching, the copper-containing organic solution being contacted, in a retraction step, with an aqueous solution of an acid so that copper passes from the organic solution to the aqueous solution, and the organic solution being recirculated from the re-extraction step to renewed extraction, characterised by the steps of passing the copper-containing acid solution obtained from the re-extraction step to a copper recovery operation, diverting a flow from the copper-containing acid solution before the operation for recovering copper from the same and adjusting the copper content of said flow so that it will be lower than the copper content of the acid solution which is used in the copper recovery operation, and recirculating said flow having an adjusted copper content to the operation for electroplating printed boards for use therein.

5. (Amended) A process as claimed in claim 1, characterised by carrying it out as a closed process, in which the plated printed board is etched with said alkaline etch bath and the acid solution from the plating is used for said re-extraction step.

6. (Amended) A process as claimed in claim 1, characterised by adjusting the copper content so that the ratio of copper content of said acid solution is  $> 0.3:1$ .

7. (Amended) A process as claimed in claim 6, characterised by adjusting the copper content so that the ratio is in the range of  $0.60:1 - 0.95:1$ .

8. (Amended) A process as claimed in claim 1, characterised by carrying out the plating in the form of pulse plating with wave-shaped pulses of current intensity.

9. (Amended) A process as claimed in claim 1, characterised by carrying out the plating in the form of pulse plating with pole reversal.

10. (Amended) A process as claimed in claim 8, characterised by carrying out the pulse plating with a pulse length of the wave-shaped pulses in the range of 1-500 ms.

11. (Amended) A process as claimed in claim 8, characterised by adjusting the period of time during which the printed board acts as cathode in the pulse plating to a value in the range of 1-200 s.

12. (Amended) A process as claimed in claim 8, characterised by adjusting the period of time during which the printed board acts as anode in the pulse plating to a value in the range of 0.1-20 s.

13. (Amended) A process as claimed in claim 8, characterised in that the maximum current intensity during the period of time when the printed board acts as cathode in the pulse plating is 10 A/dm<sup>2</sup>.

14. (Amended) A process as claimed in claim 8, characterised in that the maximum current intensity during the period of time when the printed board acts as anode in the pulse plating is 40 A/dm<sup>2</sup>.

15. (Amended) A process as claimed in claim 1, characterised by adjusting the copper content of the flow which is recirculated to the plating by the addition of acid from the re-extraction step.

16. (Amended) A process as claimed in claim 1, characterised by adjusting the copper content of the flow which is recirculated to the plating to a value in the range of 5-100 g/l.

17. (Amended) A process as claimed in claim 16, characterised by adjusting said copper content to a value in the range of 15-30 g/l.

18. (Amended) A process as claimed in claim 1, characterised by adjusting the content of anion from the used acid to a value in the range of 25-250 g/l in the flow which is used in the plating.

19. (Amended) A process as claimed in claim 1, characterised in that the content of anion from the used acid is substantially the same in the copper recovery operation as in the plating operation.

20. (Amended) A process as claimed in claim 8, characterised by carrying out the pulse plating without any additives of the kind which is used in non-pulse plating of printed boards.

21. (Amended) A process as claimed in claim 1, characterised by reducing the content of alkaline substance originating from the etch bath and/or reducing the content of organic material originating from the extraction in the flow which is recirculated to the plating before subjecting the same to said plating.

23. (Amended) A process as claimed in claim 21, characterised by carrying out said reduction (- s) by means of one or more filters and/or ultrafilters.

24. (Amended) A process as claimed in claim 1, characterised by removing colloidal copper before the plating from the flow which is recirculated to the plating.

25. (Amended) A process as claimed in claim 1, characterised by using as equipment for said extraction one or more extractors of the type in which the separation takes place by means of energy supplied from the outside.

Please add the following new Claims 26 to 43:

26. (New) A process according to claim 1 wherein said alkaline etch both is an ammoniacal etch bath.

27. (New) A process according to claim 1 wherein said acid of said aqueous solution of an acid is sulphuric acid.

28. (New) A process as claimed in claim 1, characterised by adjusting the copper content so that the ratio of copper content of said acid solution is  $>0.5:1$ .

29. (New) A process as claimed in claim 6, characterised by adjusting the copper content so that the ratio is in the range of  $0.75:1 - 0.95:1$ .

30. (New) A process as claimed in claim 1, characterised by carrying out the plating in the form of pulse plating with square pulses of current intensity.

31. (New) A process as claimed in claim 8, characterised by carrying out the pulse plating with a pulse length of the wave-shaped pulses in the range of 10-50 ms.



32. (New) A process as claimed in claim 8, characterised by adjusting the period of time during which the printed board acts as cathode in the pulse plating to a value in the range of 10-100 s.

33. (New) A process as claimed in claim 8, characterised by adjusting the period of time during which the printed board acts as anode in the pulse plating to a value in the range of 1-10 s.

34. (New) A process as claimed in claim 8, characterised in that the maximum current intensity during the period of time when the printed board acts as cathode in the pulse plating is 5 A/dm<sup>2</sup>.

35. (New) A process as claimed in claim 8, characterised in that the maximum current intensity during the period of time when the printed board acts as cathode in the pulse plating is 3 A/dm<sup>2</sup>.

36. (New) A process as claimed in claim 8, characterised in that the maximum current intensity during the period of time when the printed board acts as anode in the pulse plating is 10 A/dm<sup>2</sup>.

37. (New) A process as claimed in claim 8, characterised in that the maximum current intensity during the period of time when the printed board acts as anode in the pulse plating is 5 A/dm<sup>2</sup>.

38. (New) A process as claimed in claim 1, characterised by adjusting the copper content of the flow which is recirculated to the plating to a value in the range of 10-50 g/l.

39. (New) A process as claimed in claim 16, characterised by adjusting said copper content to a value in the range of 20-25 g/l.

40. (New) A process as claimed in claim 1, characterised by adjusting the content of anion from the used acid to a value in the range of 50-200 g/l in the flow which is used in the plating.

41. (New) A process as claimed in claim 21, characterised by carrying out said reduction (- s) by means of one or more charcoal filters and/or ultrafilters.

42. (New) A process as claimed in claim 22, characterised by carrying out said reduction (- s) by means of one or more filters and/or ultrafilters.

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Page 9

43. (New) A process as claimed in claim 1, characterised by removing colloidal copper before the plating by means of one or more ultrafilters from the flow which is recirculated to the plating.

**REMARKS**

The present Amendment provides the Abstract of the Disclosure on a separate sheet, and modifies the form only of the claims so as to eliminate the use of multiple dependency and to provide some of the originally included subject matter in separate claims.

The examination and allowance of the Application are respectfully requested.

Respectfully submitted,

BURNS, DOANE, SWECKER & MATHIS, L.L.P.

By: Benton S. Duffett Jr.  
Benton S. Duffett, Jr.  
Registration No. 22,030

P.O. Box 1404  
Alexandria, Virginia 22313-1404  
(703) 836-6620

Date: August 20, 2001

**Attachment to Preliminary Amendment dated August 20, 2001**

**Marked-up Claims -**

1. (Amended) A process for recovering copper from an alkaline[, preferably ammoniacal,] etch bath from an etching process in which printed boards electroplated with copper are etched with the alkaline etch bath and then rinsed with water, copper being removed from the alkaline etch bath by extraction with an organic solution containing a reagent, which forms with copper a complex compound, which is extracted by the organic solution, the alkaline etch bath being recirculated to renewed etching, the copper-containing organic solution being contacted, in a retraction step, with an aqueous solution of an acid[, preferably sulphuric acid,] so that copper passes from the organic solution to the aqueous solution, and the organic solution being recirculated from the re-extraction step to renewed extraction, characterised by the steps of passing the copper-containing acid solution obtained from the re-extraction step to a copper recovery operation, [preferably for producing metallic copper by electrolysis,] diverting a flow from the copper-containing acid solution before the operation for recovering copper from the same and adjusting the copper content of said flow so that it will be lower than the copper content of the acid solution which is used in the copper recovery operation, and recirculating said flow having an adjusted copper content to the operation for electroplating printed boards for use therein.

**Attachment to Preliminary Amendment dated August 20, 2001**

**Marked-up Claims -**

5. (Amended) A process as claimed in [any one of the preceding claims] claim 1, characterised by carrying it out as a closed process, in which the plated printed board is etched with said alkaline etch bath and the acid solution from the plating is used for said re-extraction step.

6. (Amended) A process as claimed in [any one of the preceding claims] claim 1, characterised by adjusting the copper content so that the ratio of copper content of said acid solution is  $> 0.3:1$ [, preferably  $> 0.5:1$ ].

7. (Amended) A process as claimed in claim 6, characterised by adjusting the copper content so that the ratio is in the range of  $0.60:1 - 0.95:1$ [, preferably  $> 0.75:1 - 0.95:1$ ].

8. (Amended) A process as claimed in [any one of the preceding claims] claim 1, characterised by carrying out the plating in the form of pulse plating with wave-shaped [, preferably square] pulses of current intensity.

9. (Amended) A process as claimed in [any one of the preceding claims] claim 1, characterised by carrying out the plating in the form of pulse plating with pole reversal.

**Attachment to Preliminary Amendment dated August 20, 2001**

**Marked-up Claims -**

10. (Amended) A process as claimed in claim 8 [or 9], characterised by carrying out the pulse plating with a pulse length of the wave-shaped pulses in the range of 1-500 ms[, preferably 10-50 ms].

11. (Amended) A process as claimed in [any one of claims 8-10] claim 8, characterised by adjusting the period of time during which the printed board acts as cathode in the pulse plating to a value in the range of 1-200 s[, preferably 10-100 s].

12. (Amended) A process as claimed in [any one of claims 8-11] claim 8, characterised by adjusting the period of time during which the printed board acts as anode in the pulse plating to a value in the range of 0.1-20 s[ preferably 1-10 s].

13. (Amended) A process as claimed in [any one of claims 8-12] claim 8, characterised in that the maximum current intensity during the period of time when the printed board acts as cathode in the pulse plating is 10 A/dm<sup>2</sup>[, preferably 5 A/dm<sup>2</sup> and most preferably 3 A/dm<sup>2</sup>].

**Attachment to Preliminary Amendment dated August 20, 2001**

**Marked-up Claims -**

14. (Amended) A process as claimed in [any one of claims 8-13] claim 8, characterised in that the maximum current intensity during the period of time when the printed board acts as anode in the pulse plating is 40 A/dm<sup>2</sup>[, preferably 10 A/dm<sup>2</sup> and most preferably 5 A/dm<sup>2</sup>].

15. (Amended) A process as claimed in [any one of the preceding claims] claim 1, characterised by adjusting the copper content of the flow which is recirculated to the plating by the addition of acid from the re-extraction step.

16. (Amended) A process as claimed in [any one of the preceding claims] claim 1, characterised by adjusting the copper content of the flow which is recirculated to the plating to a value in the range of 5-100 g/l[, preferably 10-50 g/l].

17. (Amended) A process as claimed in claim 16, characterised by adjusting said copper content to a value in the range of 15-30 g/l[, preferably 20-25 g/l].

18. (Amended) A process as claimed in [any one of the preceding claims] claim 1, characterised by adjusting the content of anion from the used acid[, preferably sulphuric acid,] to a value in the range of 25-250 g/l[, preferably 50-200 g/l,] in the flow which is used in the plating.

**Attachment to Preliminary Amendment dated August 20, 2001**

**Marked-up Claims -**

19. (Amended) A process as claimed in [any one of the preceding claims] claim 1, characterised in that the content of anion from the used acid is substantially the same in the copper recovery operation as in the plating operation.

20. (Amended) A process as claimed in [any one of claims 8-19] claim 8, characterised by carrying out the pulse plating without any additives of the kind which is used in non-pulse plating of printed boards.

21. (Amended) A process as claimed in [any one of the preceding claims] claim 1, characterised by reducing the content of alkaline substance[, preferably ammonia,] originating from the etch bath and/or reducing the content of organic material originating from the extraction in the flow which is recirculated to the plating before subjecting the same to said plating.

23. (Amended) A process as claimed in claim 21 [or 22], characterised by carrying out said reduction (- s) by means of one or more filters[, preferably charcoal filters] and/or ultrafilters.



**Attachment to Preliminary Amendment dated August 20, 2001**

**Marked-up Claims -**

24. (Amended) A process as claimed in [any one of the preceding claims] claim 1, characterised by removing colloidal copper before the plating[, preferably by means of one or more filters, in particular ultrafilters,] from the flow which is recirculated to the plating.

25. (Amended) A process as claimed in [any one of the preceding claims] claim 1, characterised by using as equipment for said extraction one or more extractors of the type in which the separation takes place by means of energy supplied from the outside.

### Abstract of the Disclosure

A process for recovering copper from an alkaline etch bath from an etching process in which printed boards plated with copper are etched with the alkaline etch bath and then rinsed with water, copper being removed by extraction with an organic solution, from which it is re-extracted in an acid solution. Said acid solution is passed to an operation for recovering copper, e.g. by electrolysis, but before said copper recovery a flow is diverted, in which the copper content is adjusted to a value below the value of the acid solution for copper recovery and which is used for the plating of printed boards.

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1005 Rec'd PATT 20 AUG 2001

Patent

Attorney's Docket No. 003300-817

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re Patent Application of )  
 )  
ROBERT PACHOLIK et al. ) **BOX PCT**  
 ) **Attn: DO/EO/US**  
Application No.: (unassigned) )  
 ) Group Art Unit: (unassigned)  
Filed: August 20, 2001 ) Examiner: (unassigned)  
 )  
For: COPPER RECOVERY PROCESS )

**TRANSMITTAL OF NEW FIG. 1**

Assistant Commissioner for Patents  
Washington, D.C. 20231


Sir:

Applicants provide a new FIG. 1 to be substituted for that originally filed in the International Application.

The new FIG. 1 is corrected to include "36" at the upper part of the drawing. Also an arrow has been deleted near reference "18". Such matters were addressed by the Examiner at the last page of the International Preliminary Examination Report.

Respectfully submitted,

BURNS, DOANE, SWECKER & MATHIS, L.L.P.

By: 

Benton S. Duffett, Jr.  
Registration No. 22,030

P.O. Box 1404  
Alexandria, Virginia 22313-1404  
(703) 836-6620

Date: August 20, 2001

09/913938

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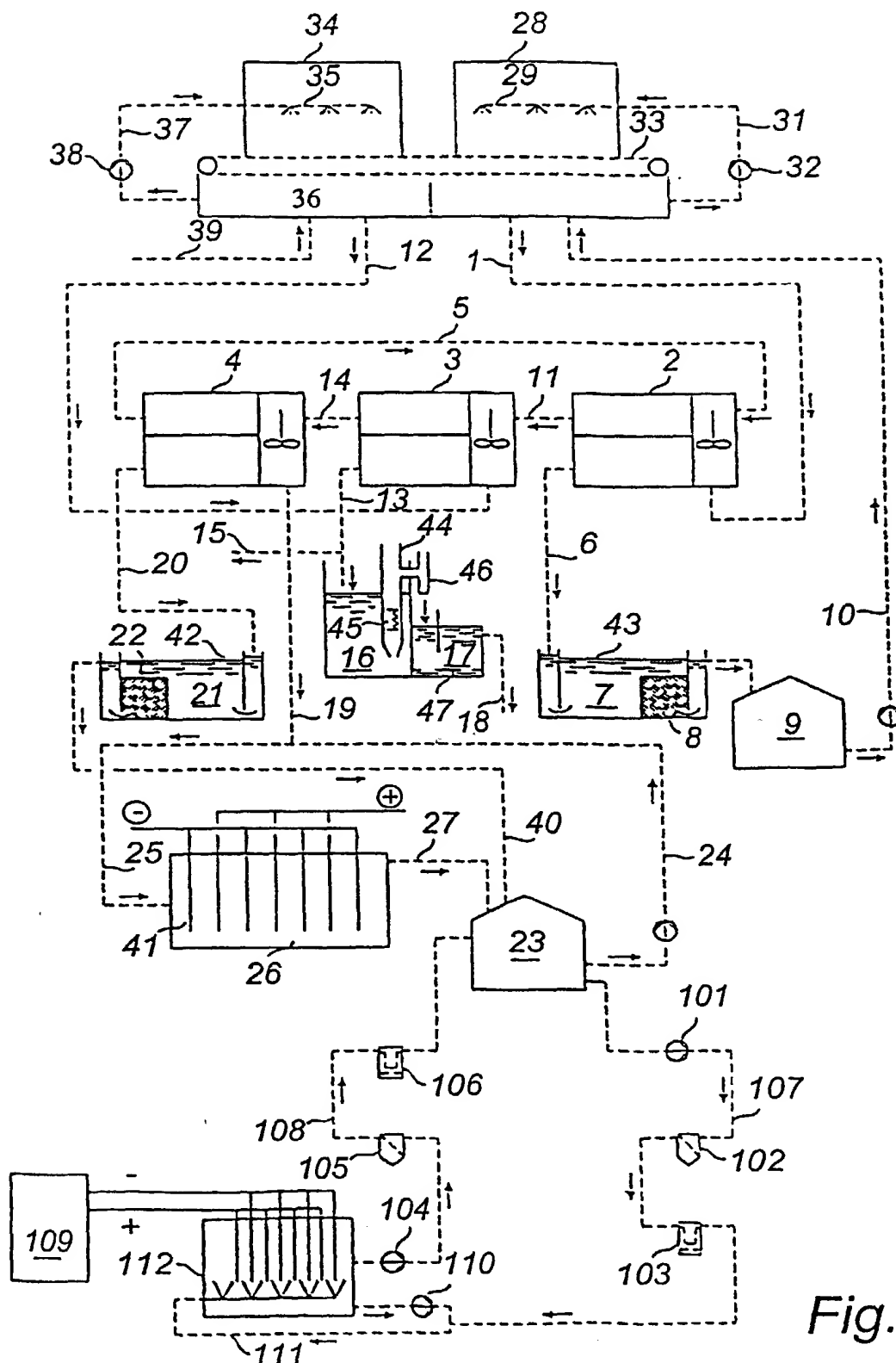


Fig. 1

COPPER RECOVERY PROCESSTechnical Field

The present invention relates to the technical field of recovering an etch bath from an etching process in which printed boards coated with copper are etched with the alkaline etch bath and then rinsed with water. The recovery of copper from such an etch bath as well as the recovery of the etch bath itself are known per se, but the invention relates to the recovery of copper in a manner that is completely new in this connection and very advantageous.

Background of the Invention

Today, electronics are primarily composed of components which are soldered onto printed boards. The manufacture of these printed boards has increased rapidly. Advanced electronics, such as computers, require very complicated printed boards and the manufacture thereof has resulted in the emergence of a special industry.

A printed board consists of a plate made of a base material, which is often a plastic material, e.g. epoxy. This plate is coated with a thin (e.g. about 15-20  $\mu\text{m}$ ) copper layer on both sides.

The manufacture of these printed boards may, for instance, be carried out in the following manner. According to a predetermined pattern, holes of different size are drilled into the plates so that ducts are formed between the two sides and so that holes are formed, in which components can be mounted. After that, all surfaces are coated with a copper layer which has a thickness of about 2-4  $\mu\text{m}$  and which is applied in a chemical manner instead of electrochemically. This is necessary since the walls of the holes consist of a material (e.g. epoxy) on which it is not possible to use electrochemical plating. As a result, an integral and continuous copper surface is ob-

tained, which can subsequently be coated by means of electrochemical plating.

The next step in the manufacture of printed boards is to make the desired conductive pattern. To this end, the surfaces which are to be free from copper are coated with a non-conductive protective film, and the boards are fed into a so-called electroplating line. This line comprises a number of different baths, into which the boards are dipped.

In one of these baths, the exposed copper surfaces are coated with additional copper to provide the desired thickness of conductors, after which the same surfaces are coated with a tin layer, which will serve as a protection during the subsequent etching.

After that, the etching of the printed boards in question takes places, which is usually carried out by means of alkaline solutions. Preferably, they are ammoniacal and contain free ammonia as well as one or more ammonium salts (e.g.  $\text{NH}_4\text{Cl}$ ,  $\text{NH}_4\text{HCO}_3$ ). When etching, copper is removed from the surfaces of the printed board, on which the copper layer is exposed to the solution. Gradually, the copper content of the bath rises to a level, on which the speed of etching drastically decreases and the bath is no longer usable. Usually, the upper limit of the copper content is about 150-170 g/l, depending on the composition of the bath. After the etching, the printed boards are rinsed with water to be clean.

The boards are then passed on to be stripped of tin and coated with varnish, wax, etc.

A method for regenerating the etch bath in a process of this kind is known from EP-B1-0005415. This method may be directly connected to the etching process and it involves in short the regeneration of the etch bath for renewed use in an etching process, copper being removed from the etch bath by extraction. The method also allows treatment of the rinsing water so that it may subsequently be let out in an environmentally suitable manner.

The copper which is extracted from the etch bath and preferably also from the rinsing water is recovered as metallic copper by electrolysis.

5 Description of the Invention

The present invention relates to a new and, in this connection, extremely advantageous process for recovering copper from an alkaline etch bath of the above-mentioned kind. According to the invention, it has thus surpris-  
10 ingly been found that it is possible not only to recover copper from the etch bath and preferably also from the rinsing water in an alternative new manner but also to reuse recovered copper in the plating process. Generally, the process according to the invention makes it possible  
15 to directly reuse a copper-containing solution which is obtained after the extraction from the alkaline etch bath in the plating of the printed boards, if a flow is diverted from the copper-containing acid solution which is obtained from the re-extraction step and the balance of  
20 the copper content is adjusted between this flow and the acid solution which is withdrawn for recovering copper, e.g. metallic copper by electrolysis. As a result, the need for cost-demanding electrolysis for recovering metallic copper is reduced, at the same time as the need  
25 for additives of expensive anode copper is reduced or eliminated in the plating. In addition, it is possible to directly and in the same place use the recovered copper which previously could only be sold at a price of about  $\frac{1}{4}$  of the price of said anode copper while achieving a  
30 plating quality which is at least equally good or even better than the quality obtained in plating according to the plating technique which was previously commercially used.

Moreover, one condition of successful plating, i.e.  
35 with a satisfactory evenness of surface and satisfactory coating in holes and the like, in connection with commercial methods previously used for electroplating has been

the addition of a number of different chemicals. Besides the fact that these chemicals to a certain extent have not been compatible with reagents which are used in the extraction, and therefore must be removed according to prior-art technique, which has complicated the process and made it more expensive, it has thus been found that the use of these chemicals may be reduced or completely eliminated in the process according to the present invention, if the plating is carried out under special conditions, in particular pulse plating. According to the invention, it has thus been found that at least equally good results as according to prior-art technique may be achieved by this new method according to the invention, which results in considerable advantages in terms of both costs and environment. Besides the fact that the process is simpler and cheaper, the process according to the invention also allows a completely closed or continuous process, in which also the plating step may be included in the prior-art etching and recovery process.

Further advantages of the invention will appear from the description below.

In this connection, it can be added that pulse plating of printed boards is known per se from Processing of Advanced Materials (1994) 9, pp 148-154, but this publication does not in any way disclose or even suggest that such plating could be integrated in a process of the kind involved in the present invention and even less so under the same circumstances and with the same results as in the case of the present invention.

Thus, the process according to the invention is a process for recovering copper from an alkaline, preferably ammoniacal, etch bath from an etching process in which printed boards electroplated with copper are etched with the alkaline etch bath and then rinsed with water, a part of the copper content of the alkaline etch bath being removed by extraction with an organic solution containing a reagent, which forms with copper a complex com-



pound, which is extracted by the organic solution, the alkaline etch bath being recirculated to renewed etching, the copper-containing organic solution being contacted, in a re-extraction step, with an aqueous solution of  
5 acid, preferably sulphuric acid, so that copper passes from the organic solution to the aqueous solution, and the organic solution being recirculated from the re-extraction step to renewed extraction. The process according to the invention is characterised by the steps of  
10 passing the copper-containing acid solution obtained from the re-extraction step to a copper recovery operation, preferably for producing metallic copper by electrolysis, diverting a flow from the copper-containing acid solution before the operation for recovering copper from the same and adjusting the copper content of said flow so that it  
15 will be lower than the copper content of the acid solution which is used in the copper recovery operation, and re-circulating said flow having an adjusted copper content to the operation for electroplating printed boards  
20 for use therein.

According to a preferred embodiment of the process, copper is also removed from the rinsing water from the etching process by extraction with an organic solution containing a reagent, which forms with copper a complex  
25 compound which is extracted by the organic solution.

One embodiment of the last-mentioned process involves using the same organic solution for the rinsing water as for the alkaline etch bath and first removing copper from the alkaline etch bath, then contacting the  
30 thus obtained copper-containing organic solution with the rinsing water and subsequently subjecting the organic solution to said re-extraction.

Another embodiment of said process involves using the same organic solution for the rinsing water as for  
35 the alkaline etch bath and first removing copper from the rinsing water, then contacting the thus obtained copper-containing organic solution with the alkaline etch bath

and subsequently subjecting the organic solution to said re-extraction.

A particularly preferred embodiment of the process according to the invention implies that it is carried out  
5 as a closed process, in which the plated printed board is etched with said alkaline etch bath and the acid solution from the plating is used for said re-extraction step. Most preferably, also the acid solution from the copper recovery operation is recirculated to the re-extraction  
10 step or steps.

According to the invention, it has been found that excellent results are achieved if the flow is adjusted so that the ratio of copper content of said flow:copper content of said acid solution is  $> 0.3:1$ , preferably  $> 0.5:1$ .  
15 According to a particularly preferred embodiment of the process according to the invention, the flow is adjusted so that said ratio is in the range of  $0.60:1-0.95:1$ , most preferably  $0.75:1-0.95:1$ .

As suggested above, a particularly advantageous embodiment of the process implies that the plating is carried out in the form of pulse plating. In this connection, pulse plating refers to plating with pole reversal and/or pulses varying for current intensity. This can be achieved with suitable rectifier equipment. The varying  
20 pulses and/or the pole reversal may be regularly or irregularly wave-shaped, suitably square. As concerns this technique as such, information may be retrieved from the above-mentioned publication on pulse plating in general, but according to the invention it has been found that advantageous results are achieved under special conditions.  
25 These conditions may be summarised as follows.

The pulse plating is preferably carried out with a pulse length of the wave-shaped pulses in the range of 1-500 ms, preferably 10-50 ms. In the case of these pulses,  
35 it is possible to have pulse lengths varying within the indicated ranges or use substantially the same, or exactly the same, pulse length for pulse peaks as for pulse

bases, i.e. the period of time during which the wave-shaped, preferably square, pulses have maximum and minimum current intensity, respectively, alternatively maximum current intensity for +-voltage and -voltage, respectively.

Preferably, the period of time during which the printed board acts as cathode in the pulse plating is adjusted to a value in the range of 1-200 s, in particular 10-100 s.

The period of time during which the printed board acts as anode in the pulse plating is preferably adjusted to a value in the range of 0.1-20 s, in particular 1-10 s.

The maximum current intensity during the period of time when the printed board acts as cathode in the pulse plating is suitably 10 A/dm<sup>2</sup>, preferably 5 A/dm<sup>2</sup> and most preferably 3 A/dm<sup>2</sup>.

The maximum current intensity during the period of time when the printed board acts as anode in the pulse plating is preferably 40 A/dm<sup>2</sup>, more preferably 10 A/dm<sup>2</sup> and most preferably 5 A/dm<sup>2</sup>.

Generally, the average current intensity when the printed board acts as anode is greater than the average current intensity when the printed board acts as cathode.

According to the invention, it has furthermore been found that excellent results are achieved if the copper content of the flow which is recirculated to the plating is adjusted to a value in the range of 5-100 g/l, preferably 10-50 g/l, more preferably 15-30 g/l and most preferably 20-25 g/l.

In the case of such an adjustment, it has been found that an adjustment of the copper content of the flow which is recirculated to the plating functions extremely well by the addition of acid from the re-extraction step.

As a rule, it is preferably the same order of magnitude of the copper content of the acid solution which is used in the copper recovery operation, use being made,

Among other factors having an influence on the plating, mention can be made of the content of alkaline substance originating from the etch bath and the content of organic material originating from the extraction. According to the invention, it has thus been found that excellent results may be achieved in the process according

to the invention if the content of said alkaline substance and/or the content of said organic material in the flow recirculated to the pulse plating is reduced, and preferably eliminated, before plating.

5 This reduction or these reductions may be carried out at different stages in the process according to the invention, but a particularly preferred embodiment is represented by the case where the reduction(-s) in question is/are carried out by means of one or more separate  
10 water washing steps in connection with the equipment which is used for the extraction. Such water washing steps are preferably integrated before the last step of the extraction process.

The reduction(-s) in question may also be carried  
15 out by means of one or more filters, preferably filters of charcoal and/or ultrafilter type. Such filters may also be placed at various locations in the process, but they are advantageously positioned in the separate loop which is represented by the flow which is recirculated to  
20 the plating.

Another advantageous embodiment of the process according to the invention is represented by the case in which colloidal copper is removed before the plating, preferably by means of one or more filters, in particular  
25 ultrafilters, from the flow which is recirculated to the pulse plating.

As will be described in more detail below in connection with the embodiments shown in the drawings, mixer-settler devices have been used in prior-art extraction.  
30 According to the invention, it has, however, been found that particularly favourable results in the plating may be achieved if these mixer-settler devices are wholly or partly replaced by one or more extractors. Such extractors are, of course, known per se, but they have been  
35 found to yield advantageous results in connection with the invention. By extractor is essentially meant a device in which the above-mentioned settler in the mixer-settler

In the second extraction step 3, the organic extraction solution which is supplied from the mixer-settler 2

via a conduit 11 is contacted with washing water supplied from the container 36 via a conduit 12.

The organic solution is supplied from the mixer-settler 3 via a conduit 14. The washing water is supplied from said mixer-settler 3 via a conduit 13. Sodium hydroxide is added to the washing water via a conduit 15, and the obtained alkaline water is passed into a container 16 including a vertical pipe 44. This pipe 44 has open ends at the top and the bottom and contains a heating element 45, by means of which the water in the pipe is made to boil. The pipe 44 is provided with a branch pipe 46, which extends into a sedimentation container 17. Ammonia is expelled from the boiling water in the pipe 44, which results in precipitation of copper hydroxide. The boiling results in a raised water level in the pipe 44, which makes water flow over into the sedimentation container 17 via the branch pipe 46. The precipitated copper hydroxide forms a layer 47 at the bottom of the sedimentation container 17. Water can flow out of the sedimentation container 17 via a conduit 18.

The organic solution which is fed to the mixer-settler 4 in question through the conduit 14 is contacted with a solution of sulphuric acid, which is supplied via a conduit 19. The acid solution is withdrawn via a conduit 20.

The organic solution from the re-extraction is re-circulated via the conduit 5 to the step for extraction of copper.

The solution of sulphuric acid is passed via the conduit 20 to a flotation tank 21 containing a filter 22 (activated carbon). Droplets of organic solution which have been separated from the water-based liquid form a surface layer 42. The last rests of organic liquid are removed in the filter 22. The solution is passed via a conduit 40 to a storage or buffer tank 23.

From this tank 23, a part of the copper-containing acid solution obtained from the re-extraction step is

pumped through the conduits 24 and 25 to an electrolytic cell 26, in which copper is recovered electrolytically on titanium cathodes 41. The electrolyte 27 is recirculated to the tank 23. A part of the solution which is pumped  
5 through the conduit 24 is recirculated via a conduit 19 to the re-extraction step 4.

A second part of the solution from the tank 23 is pumped by means of the pump 101 via the conduit 107 as well as via a particle filter 102 and a charcoal filter  
10 103 to a plating cell 112. 111 represents the acid which is used for the plating, whereas 110 represents a pump for circulation of the acid used for the plating.

The plating cell 112 is controlled by a rectifier 109 with pulsation and pole reversal. From the plating  
15 cell 112, the used acid solution is pumped via a pump 104, which is controlled by the level during the plating, and through the conduit 108 back to the storage tank 23. While being pumped, the solution passes a particle filter 105 and a charcoal filter 106.

20 As a rule, the copper content of the plating circuit is sensed and electrolyte from the electrolysis circuit is dosed to the plating as said copper content falls below a predetermined value.

The alternative embodiment of the mixer-settler installation 2-4 in Fig. 1, which is shown in Fig. 2 and in which the reference numerals that are common to those of the installation shown in Fig. 1 have been maintained, comprises as an additional step a separate step 3A for washing with water, so that in the flow which is subse-  
30 quently diverted for plating, the amount of substances or material originating from the etch bath and possibly also the extraction will be reduced. Otherwise, this mixer-settler device is intended to function as the device shown in Fig. 1.





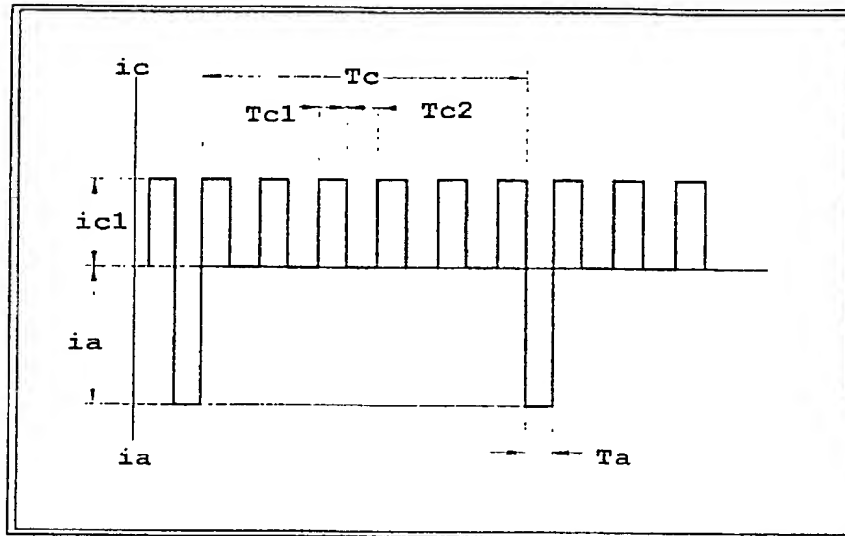
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Another qualitative advantage due to the omission of additives or chemicals in the plating is that there will be no such impurities in used copper, which has a favourable effect on the conductivity of copper.

- 5 Yet another qualitative advantage is that it is very easy to adjust the process to different types of printed boards by modifying the settings of the rectifier. This is not possible when using chemicals, since in that case large volumes of liquid have to be adjusted.

**Table 1****Chemical specification**

Copper content	Acid content	Chloride content	Temperature	Air flow	Circulation
20.5 g/l	145 g/l	40 ppm	23 degr.C	25 l/min	300 l/h

**Panel specification**

Thickness	Base copper	Chem.copper	Hole diameter
1.6 mm	17 microns	6 microns	0.8 mm

5

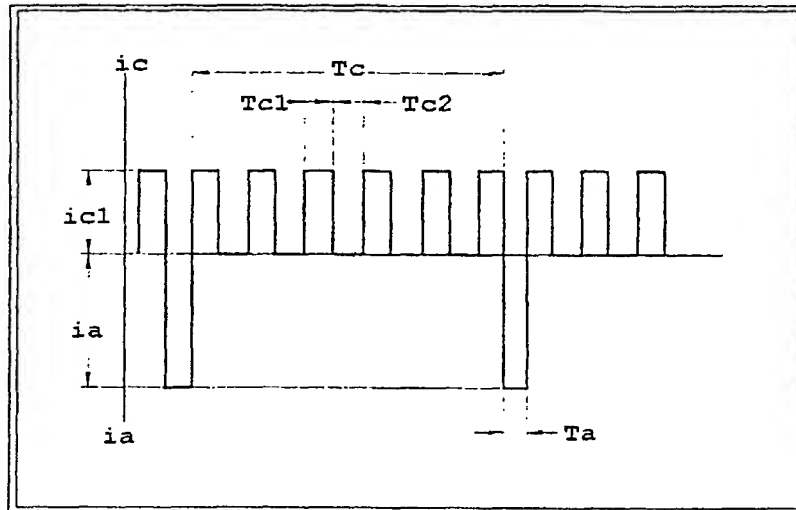
**Electric specification**

Panel No.	ic(A/dm <sup>2</sup> )	ia(A/dm <sup>2</sup> )	Tc (sec)	Tc1(msec)	Tc2(msec)	Ta(sec)
1	3	4.5	60	20	20	8
2	3	4.5	20	20	20	8
3	3	4.5	20	40	40	8
4	2	3	60	20	20	8
5	2	3	20	20	20	8
6	2	3	20	40	40	8

**Results**

Panel No.	Qa/Qc	Hole-Surface ratio	Tc+Ta	Strain	Remarks
1	0.2	1.0	68	41	
2	0.6	1.4	28	40	
3	0.6	1.5	28	44	
4	0.2	1.0	68	42	
5	0.6	1.8	28	42	
6	0.6	1.7	28	46	

**Table 2**



**Chemical specification**

Copper content	Acid content	Chloride content	Temperature	Air flow	Circulation
20.5 g/l	145 g/l	40 ppm	23 degr.C	25 l/min	300 l/h

**Panel specification**

Thickness	Base copper	Chem.copper	Hole diameter
1.6 mm	17 microns	6 microns	0.4 mm

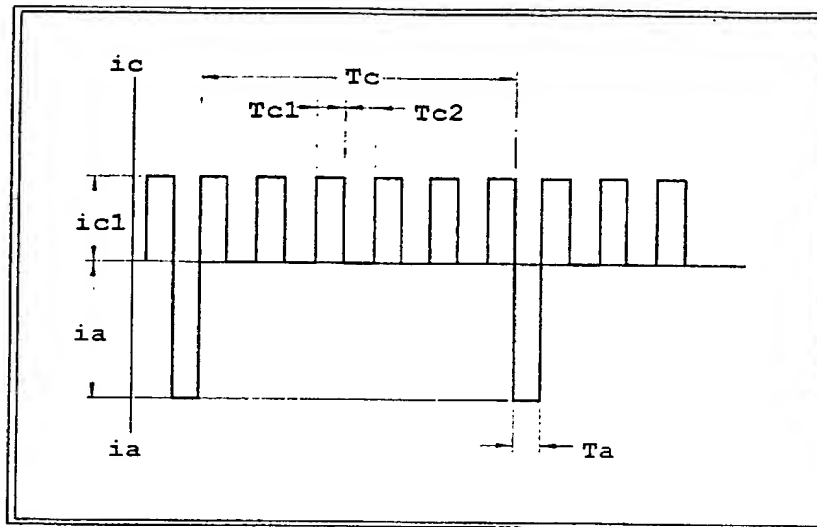
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**Electric specification**

Panel No.	ic(A/dm <sup>2</sup> )	ia(A/dm <sup>2</sup> )	Tc (sec)	Tc1(msec)	Tc2(msec)	Ta(sec)
1	3	4.5	60	20	20	8
2	3	4.5	20	20	20	8
3	3	4.5	20	40	40	8
4	2	3	60	20	20	8
5	2	3	20	20	20	8
6	2	3	20	40	40	8

**Results**

Panel No.	Qa/Qc	Hole-Surface ratio	Tc+Ta	Strain	Remarks
1	0.2	1.3	68	40	
2	0.6	2.6	28	40	
3	0.6	2.7	28	43	
4	0.2	1.5	68	42	
5	0.6	3.0	28	41	
6	0.6	2.8	28	47	

**Table 3****Chemical specification**

Copper content	Acid content	Chloride content	Temperature	Air flow	Circulation
22.5 g/l	140 g/l	40 ppm	23 degr.C	25 l/min	300 l/h

**5 Panel specification**

Thickness	Base copper	Chem.copper	Hole diameter
1.6 mm	17 microns	6 microns	0.8 mm

**Electric specification**

Panel No.	ic(A/dm <sup>2</sup> )	ia(A/dm <sup>2</sup> )	Tc (sec)	Tc1(msec)	Tc2(msec)	Ta(sec)
1	3	4.5	60	20	20	8
2	3	4.5	20	20	20	8
3	3	4.5	20	40	40	8
4	2	3	60	20	20	8
5	2	3	20	20	20	8
6	2	3	20	40	40	8

**Results**

Panel No.	Qa/Qc	Hole-Surface ratio	Tc+Ta	Strain	Remarks
1	0.2	0.9	68	39	
2	0.6	1.4	28	39	
3	0.6	1.4	28	42	
4	0.2	1.1	68	40	
5	0.6	1.8	28	41	
6	0.6	1.6	28	43	

## CLAIMS

1. A process for recovering copper from an alkaline, preferably ammoniacal, etch bath from an etching process  
5 in which printed boards electroplated with copper are etched with the alkaline etch bath and then rinsed with water, copper being removed from the alkaline etch bath by extraction with an organic solution containing a reagent, which forms with copper a complex compound, which  
10 is extracted by the organic solution, the alkaline etch bath being recirculated to renewed etching, the copper-containing organic solution being contacted, in a re-extraction step, with an aqueous solution of an acid, preferably sulphuric acid, so that copper passes from the  
15 organic solution to the aqueous solution, and the organic solution being recirculated from the re-extraction step to renewed extraction, c h a r a c t e r i s e d by the steps of passing the copper-containing acid solution obtained from the re-extraction step to a copper recovery  
20 operation, preferably for producing metallic copper by electrolysis, diverting a flow from the copper-containing acid solution before the operation for recovering copper from the same and adjusting the copper content of said flow so that it will be lower than the copper content of  
25 the acid solution which is used in the copper recovery operation, and recirculating said flow having an adjusted copper content to the operation for electroplating printed boards for use therein.

2. A process as claimed in claim 1, c h a r a c -  
30 t e r i s e d by also removing copper from the rinsing water from the etching process by extraction with an organic solution containing a reagent, which forms with copper a complex compound which is extracted by the organic solution.

35 3. A process as claimed in claim 2, c h a r a c -  
t e r i s e d by using the same organic solution for the rinsing water as for the alkaline etch bath and first re-

moving copper from the alkaline etch bath, then contacting the thus obtained copper-containing organic solution with the rinsing water and subsequently subjecting the organic solution to said re-extraction.

5           4. A process as claimed in claim 1, characterised by using the same organic solution for the rinsing water as for the alkaline etch bath and first removing copper from the rinsing water, then contacting the thus obtained copper-containing organic solution with the  
10 alkaline etch bath and subsequently subjecting the organic solution to said re-extraction.

          5. A process as claimed in any one of the preceding claims, characterised by carrying it out as a closed process, in which the plated printed board is  
15 etched with said alkaline etch bath and the acid solution from the plating is used for said re-extraction step.

          6. A process as claimed in any one of the preceding claims, characterised by adjusting the copper content so that the ratio of copper content of said  
20 flow:copper content of said acid solution is  $> 0.3:1$ , preferably  $> 0.5:1$ .

          7. A process as claimed in claim 6, characterised by adjusting the copper content so that the ratio is in the range of  $0.60:1 - 0.95:1$ , preferably  
25  $0.75:1 - 0.95:1$ .

          8. A process as claimed in any one of the preceding claims, characterised by carrying out the plating in the form of pulse plating with wave-shaped, preferably square pulses of current intensity.

30           9. A process as claimed in any one of the preceding claims, characterised by carrying out the plating in the form of pulse plating with pole reversal.

          10. A process as claimed in claim 8 or 9, characterised by carrying out the pulse plating with  
35 a pulse length of the wave-shaped pulses in the range of 1-500 ms, preferably 10-50 ms.

11. A process as claimed in any one of claims 8-10, characterised by adjusting the period of time during which the printed board acts as cathode in the pulse plating to a value in the range of 1-200 s, preferably 10-100 s.

12. A process as claimed in any one of claims 8-11, characterised by adjusting the period of time during which the printed board acts as anode in the pulse plating to a value in the range of 0.1-20 s, preferably 1-10 s.

13. A process as claimed in any one of claims 8-12, characterised in that the maximum current intensity during the period of time when the printed board acts as cathode in the pulse plating is 10 A/dm<sup>2</sup>, preferably 5 A/dm<sup>2</sup> and most preferably 3 A/dm<sup>2</sup>.

14. A process as claimed in any one of claims 8-13, characterised in that the maximum current intensity during the period of time when the printed board acts as anode in the pulse plating is 40 A/dm<sup>2</sup>, preferably 10 A/dm<sup>2</sup> and most preferably 5 A/dm<sup>2</sup>.

15. A process as claimed in any one of the preceding claims, characterised by adjusting the copper content of the flow which is recirculated to the plating by the addition of acid from the re-extraction step.

16. A process as claimed in any one of the preceding claims, characterised by adjusting the copper content of the flow which is recirculated to the plating to a value in the range of 5-100 g/l, preferably 10-50 g/l.

17. A process as claimed in claim 16, characterised by adjusting said copper content to a value in the range of 15-30 g/l, preferably 20-25 g/l.

18. A process as claimed in any one of the preceding claims, characterised by adjusting the content of anion from the used acid, preferably sulphuric acid, to a value in the range of 25-250 g/l, preferably 50-200 g/l, in the flow which is used in the plating.





Abstract of the Disclosure

A process for recovering copper from an alkaline etch bath from an etching process in which printed boards plated with copper are etched with the alkaline etch bath and then rinsed with water, copper being removed by extraction with an organic solution, from which it is re-extracted in an acid solution. Said acid solution is passed to an operation for recovering copper, e.g. by electrolysis, but before said copper recovery a flow is diverted, in which the copper content is adjusted to a value below the value of the acid solution for copper recovery and which is used for the plating of printed boards.

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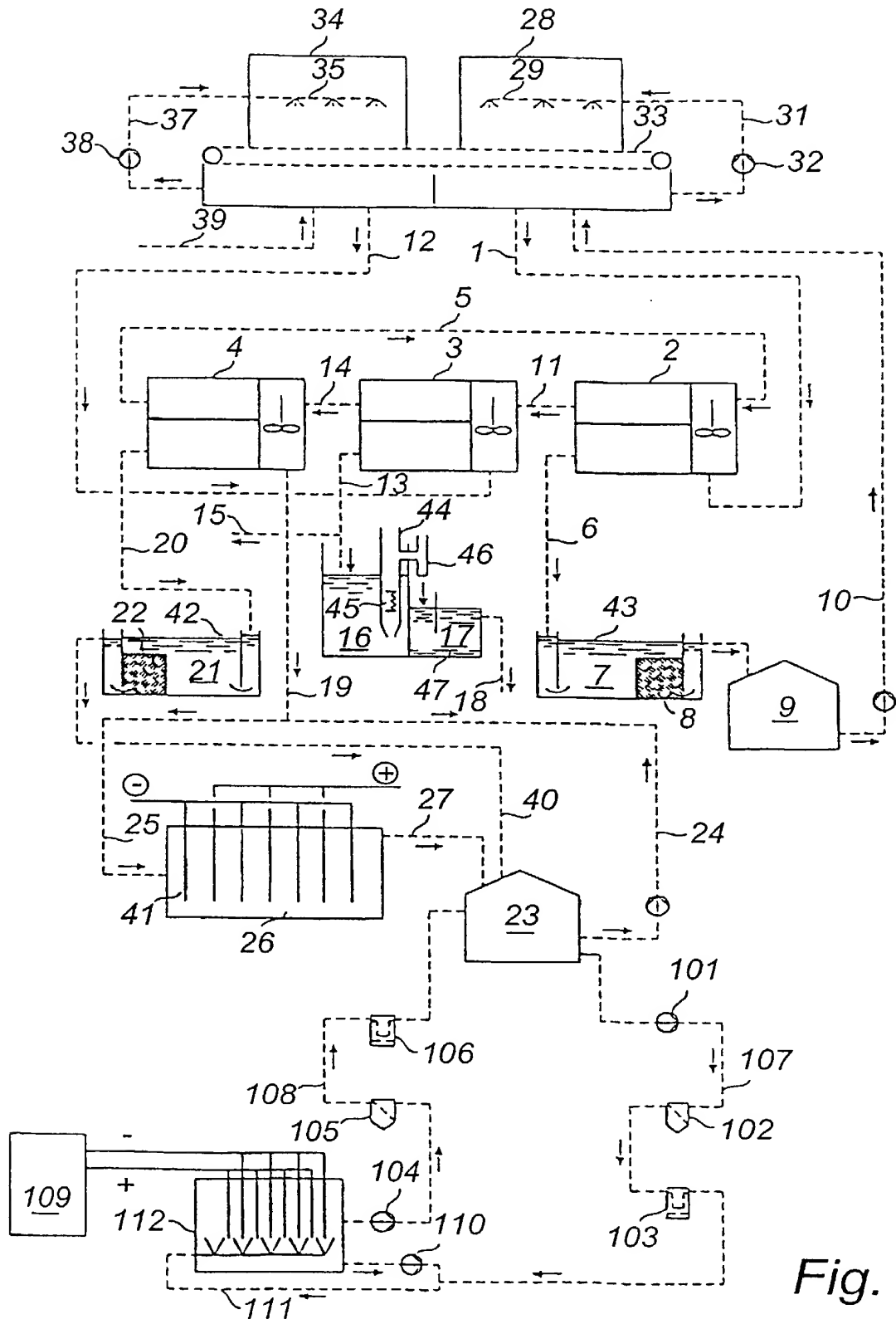
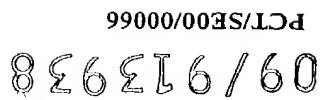


Fig. 1



**COMBINED DECLARATION AND POWER OF ATTORNEY  
FOR UTILITY PATENT APPLICATION**

Attorney's Docket No.

003300-817

As a below-named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name;

I BELIEVE I AM THE ORIGINAL, FIRST AND SOLE INVENTOR (if only one name is listed below) OR AN ORIGINAL, FIRST AND JOINT INVENTOR (if more than one name is listed below) OF THE SUBJECT MATTER WHICH IS CLAIMED AND FOR WHICH A PATENT IS SOUGHT ON THE INVENTION ENTITLED:

COPPER RECOVERY PROCESS

the specification of which

(check one)

☐

is attached hereto;

☒

was filed on January 14, 2000 as

Application No. PCT/SE00/00066

and was amended on \_\_\_\_\_;  
(if applicable)

I HAVE REVIEWED AND UNDERSTAND THE CONTENTS OF THE ABOVE-IDENTIFIED SPECIFICATION, INCLUDING THE CLAIMS, AS AMENDED BY ANY AMENDMENT REFERRED TO ABOVE;

I ACKNOWLEDGE THE DUTY TO DISCLOSE TO THE OFFICE ALL INFORMATION KNOWN TO ME TO BE MATERIAL TO PATENTABILITY AS DEFINED IN TITLE 37, CODE OF FEDERAL REGULATIONS, Sec. 1.56 (as amended effective March 16, 1992);

I do not know and do not believe the said invention was ever known or used in the United States of America before my or our invention thereof, or patented or described in any printed publication in any country before my or our invention thereof or more than one year prior to said application; that said invention was not in public use or on sale in the United States of America more than one year prior to said application; that said invention has not been patented or made the subject of an inventor's certificate issued before the date of said application in any country foreign to the United States of America on any application filed by me or my legal representatives or assigns more than twelve months prior to said application;

I hereby claim foreign priority benefits under Title 35, United States Code Sec. 119 and/or Sec. 365 of any foreign application(s) for patent or inventor's certificate as indicated below and have also identified below any foreign application for patent or inventor's certificate on this invention having a filing date before that of the application(s) on which priority is claimed:

<b>COMBINED DECLARATION AND POWER OF ATTORNEY</b>	Attorney's Docket No. 003300-817
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COUNTRY/INTERNATIONAL	APPLICATION NUMBER	DATE OF FILING (day, month, year)	PRIORITY CLAIMED
Sweden	9900748-6	2 March 1999	YES <input checked="" type="checkbox"/> NO <input type="checkbox"/>
			YES <input type="checkbox"/> NO <input type="checkbox"/>

I hereby appoint the following attorneys and agent(s) to prosecute said application and to transact all business in the Patent and Trademark Office connected therewith and to file, prosecute and to transact all business in connection with international applications directed to said invention:

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Ronald L. Grudziecki	24,970	T. Gene Dillahunty	25,423	Brian P. O'Shaughnessy	32,747
Frederick G. Michaud, Jr.	26,003	Patrick C. Keane	32,858	Kenneth B. Leffler	36,075
Alan E. Kopecki	25,813	B. Jefferson Boggs, Jr.	32,344	Fred W. Hathaway	32,236
Regis E. Slutter	26,999	William H. Benz	25,952	Wendi L. Weinstein	34,456
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Robert G. Mukai	28,531	Richard J. McGrath	29,195		
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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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**COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY**  
(Includes Reference to Provisional and International (PCT) Applications)Attorney's Docket No.  
003300-817

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name;

I BELIEVE I AM THE ORIGINAL, FIRST AND SOLE INVENTOR (IF ONLY ONE NAME IS LISTED BELOW) OR AN ORIGINAL, FIRST AND JOINT INVENTOR (IF PLURAL NAMES ARE LISTED BELOW) OF THE SUBJECT MATTER WHICH IS CLAIMED AND FOR WHICH A PATENT IS SOUGHT ON THE INVENTION ENTITLED:

The specification of which (check only one item below):

- ☐ is attached hereto.
- ☒ was filed as United States Patent Application Number \_\_\_\_\_  
on \_\_\_\_\_  
and was amended on \_\_\_\_\_ (if applicable).
- ☐ was filed as International (PCT) Application Number \_\_\_\_\_  
on \_\_\_\_\_  
and was amended on \_\_\_\_\_ (if applicable).

I HAVE REVIEWED AND UNDERSTAND THE CONTENTS OF THE ABOVE-IDENTIFIED SPECIFICATION, INCLUDING THE CLAIMS, AS AMENDED BY ANY AMENDMENT REFERRED TO ABOVE.

I ACKNOWLEDGE THE DUTY TO DISCLOSE TO THE U.S. PATENT AND TRADEMARK OFFICE ALL INFORMATION KNOWN TO ME TO BE MATERIAL TO PATENTABILITY AS DEFINED IN TITLE 37, CODE OF FEDERAL REGULATIONS, Sec. 1.56 (as amended effective March 16, 1992);

I do not know and do not believe the said invention was ever known or used in the United States of America before my or our invention thereof, or patented or described in any printed publication in any country before my or our invention thereof or more than one year prior to said application; that said invention was not in public use or on sale in the United States of America more than one year prior to said application; that said invention has not been patented or made the subject of an inventor's certificate issued before the date of said application in any country foreign to the United States of America on any application filed by me or my legal representatives or assigns more than six months prior to said application;

I hereby claim foreign priority benefits under Title 35, United States Code, §§ 119 (a)-(e) of any foreign application(s) for patent or inventor's certificate or of any International (PCT) Application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT International (PCT) Application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

**PRIOR FOREIGN/PCT APPLICATION(S) AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. §119:**

COUNTRY (If PCT, indicate "PCT")	APPLICATION NUMBER	DATE OF FILING (day, month, year)	PRIORITY CLAIMED UNDER 35 U.S.C. §119
SE	9900748-6	02/03/99	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No
			<input type="checkbox"/> Yes <input type="checkbox"/> No
			<input type="checkbox"/> Yes <input type="checkbox"/> No
			<input type="checkbox"/> Yes <input type="checkbox"/> No
			<input type="checkbox"/> Yes <input type="checkbox"/> No

I hereby claim the benefit under Title 35, United States Code § 119(e) of any United States provisional application(s) listed below.

(APPLICATION NUMBER)

(FILING DATE)

(APPLICATION NUMBER)

(FILING DATE)

**COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY (CONT'D)**  
(Includes Reference to Provisional and International (PCT) Applications)Attorney's Docket  
No. 003300-817

I hereby claim the benefit under Title 35, United States Code, § 120 of any United States applications(s) or International (PCT) Application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that/those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, § 112, I acknowledge the duty to disclose to the U.S. Patent and Trademark Office all information known to me to be material to the patentability as defined in Title 37, Code of Federal Regulations § 1.56, which became available between the filing date of the prior application(s) and the national or international filing date of this application:

**PRIOR U.S. APPLICATIONS OR INTERNATIONAL (PCT) APPLICATIONS DESIGNATING THE U.S. FOR BENEFIT UNDER 35 U.S.C. § 120:**

U.S. APPLICATIONS		STATUS (check one)		
U.S. APPLICATION NUMBER	U.S. FILING DATE	PATENTED	PENDING	ABANDONED
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
PCT APPLICATIONS DESIGNATING THE U.S.				
PCT APPLICATION NO.	PCT FILING DATE	U.S. APPLICATION NUMBERS ASSIGNED (if any)		
PCT	14/01/00			

I hereby appoint the following attorneys and agent(s) to prosecute said application and to transact all business in the U.S. Patent and Trademark Office connected therewith and to file, prosecute and to transact all business in connection with international applications directed to said invention:

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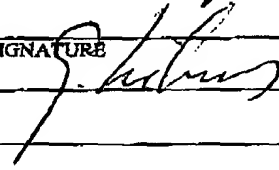


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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.



COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY (CONT'D) (Includes Reference to Provisional and International (PCT) Applications)		Attorney's Docket No. 003300-817
1-00 FULL NAME OF SOLE OR FIRST INVENTOR Robert PACHOLIK		SIGNATURE  DATE 2001.08.22
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2-00 FULL NAME OF SECOND JOINT INVENTOR, IF ANY Gunnar LIDMER		SIGNATURE  DATE 2001.08.22
RESIDENCE (CITY & STATE/COUNTRY) VALLDA, Sweden SEX		CITIZENSHIP SE
POST OFFICE ADDRESS (HOME ADDRESS) Ångås 32-35, SE-434 93 VALLDA, Sweden		
FULL NAME OF THIRD JOINT INVENTOR, IF ANY		SIGNATURE
RESIDENCE (CITY & STATE/COUNTRY)		DATE
POST OFFICE ADDRESS (HOME ADDRESS)		CITIZENSHIP
FULL NAME OF FOURTH JOINT INVENTOR, IF ANY		SIGNATURE
RESIDENCE (CITY & STATE/COUNTRY)		DATE
POST OFFICE ADDRESS (HOME ADDRESS)		CITIZENSHIP
FULL NAME OF FIFTH JOINT INVENTOR, IF ANY		SIGNATURE
RESIDENCE (CITY & STATE/COUNTRY)		DATE
POST OFFICE ADDRESS (HOME ADDRESS)		CITIZENSHIP
FULL NAME OF SIXTH JOINT INVENTOR, IF ANY		SIGNATURE
RESIDENCE (CITY & STATE/COUNTRY)		DATE
POST OFFICE ADDRESS (HOME ADDRESS)		CITIZENSHIP
FULL NAME OF SEVENTH JOINT INVENTOR, IF ANY		SIGNATURE
RESIDENCE (CITY & STATE/COUNTRY)		DATE
POST OFFICE ADDRESS (HOME ADDRESS)		CITIZENSHIP
FULL NAME OF EIGHTH JOINT INVENTOR, IF ANY		SIGNATURE
RESIDENCE (CITY & STATE/COUNTRY)		DATE
POST OFFICE ADDRESS (HOME ADDRESS)		CITIZENSHIP
FULL NAME OF NINTH JOINT INVENTOR, IF ANY		SIGNATURE
RESIDENCE (CITY & STATE/COUNTRY)		DATE
POST OFFICE ADDRESS (HOME ADDRESS)		CITIZENSHIP
FULL NAME OF TENTH JOINT INVENTOR, IF ANY		SIGNATURE
RESIDENCE (CITY & STATE/COUNTRY)		DATE
POST OFFICE ADDRESS (HOME ADDRESS)		CITIZENSHIP